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OTC REPORT 7715

IDENTIFICATION AND QUANTITATION OF PHENOLS AND ACIDS IN THUNDER BAY AND THE ST. MARY'S RIVER

by

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September, 1977.

INTRODUCTION

As part of a Great Lakes Survey for organic compounds that impart taste and odour problems, two sets of samples were received from the Water Resources Branch (Great Lakes Studies). These were from Thunder Bay, in the vicinity of the Abitibi Power and Paper Mill, and from the St. Mary's River in the vicinity of the Algoma Pulp and Paper Mill.

SAMPLES

Thunder Bay Set

Your Identification	Lab. Number
A	OMS - 630
B	- 631
C	- 632
D	- 633
E	- 634
F	- 635
G	- 636
H	- 637
I	- 638
J	- 639

St. Mary's River Set

11199	OX - 22
11201	- 23
11203	- 24
11205	- 25
11208	- 26

Your identification	Lab. Number
11210	OX - 27
11212	- 28
11233	- 29
11234	- 30
11237	- 31
11239	- 32

EXPERIMENTAL

(a) Extraction

The 500 mL samples were made basic (pH>10) with NaOH, then extracted three times with 25 mL portions of ether. The aqueous portion was then acidified with conc. HCl (pH<2), and NaCl was added until the solution was saturated. This solution was extracted four times with 25 mL portions of ether. The ethereal extracts were dried (Na_2SO_4) and then concentrated to 0.1 mL.

(b) Instrumentation

Dupont GC-MS System Model 21-491B.

GC - Varian 2740 (modified)

Column:	1.8 m x 2 mm ID. glass.
Packing:	10% SP 216 on 100/120 Supelcoport.
Carrier:	Helium 21 mls/min.
Temperature:	(a) Injection 190°C.
	(b) Detection 250°C.
	(c) Oven - program (i) 70°C hold 4 mins.
	(ii) 70-190°C at 4°/min. and hold.

Interface

Glass splitter - split ratio 3:1 FID/MS (in detector oven).

All glass transfer line at 240°C.

Jet Separator - single stage - glass at 240°C.

Mass Spectrometer

Electron impact mode - 70 ev
Source temperature - 200°C.

RESULTS AND DISCUSSION

The basic extraction allowed easy separation of the phenolic and acidic compounds from the neutral and basic organics, which greatly simplified the analysis.

In Tables 1 and 2 the phenols and acids are listed. In both Tables, only the phenols have been quantitated. The concentration is in µg/L.

The acids when present are indicated by d (detected). The detection limit for the acids and phenols was approximately 0.1 µg/L.

TABLE 1

GREAT LAKES SURVEY - ST. MARY'S RIVER

Concentration µg/L

	Sample Number OX -										
Phenols	22	23	24	25	26	27	28	29	30	31	32
Phenol	1	17	9	7	1	3	3		4	4	2
p-cresol		1	<1	<1		<1	<1		1		
Dichlorophenol											
Xylenol											
Eugenol											
Trichlorophenol											
Hyroxybenzoic acid						1	2				
2,6-Dimethyoxy-4-propenyl phenol											
Acids											
Lauric acid	d	d	d	d				d			
Myristic acid	d	d						d			
Benzoic acid						d			d		d
Furoic acid											
Palmitic acid	d							d	d	d	d
Stearic acid								d	d	d	d

TABLE 2

GREAT LAKES SURVEY - THUNDER BAY (LAKE SUPERIOR)

Concentration µg/L

	Sample Numbers OMS -									
Phenols	630	631	632	633	634	635	636	637	638	639
Phenol	36	10	8	9	3	9	4		3	1
p-Cresol	4	3	1	2	1	1	<1			
Dichlorophenol	4									
Xylenol				3		2				
Eugenol				3		4			1	
Trichlorophenol	23					3				
Hydroxybenzoic acid										
2,6-Dimethyoxy-4-propenyl phenol	24					3				
Acids										
Lauric acid										
Myristic acid										
Benzoic acid										
Furoic acid	d	d	d	d	d	d	d		d	
Palmitic acid		d	d	d	d	d	d			
Stearic acid	d									